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Green Synthesis of *Psidium guajava* Capped Hydroxyapatite Nanoparticles

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ABSTRACT

The current research has been carried out in the synthesis and characterization of hydroxyapatite nanoparticles of 26 ±5 nm from Psidium guajava leaf extract with control over dimension and composition. The reaction occurs very rapidly as the formation of spherical nanoparticles is almost completed within 30nm. The probable pathway of the green synthesis is suggested. Appearance, crystalline nature, size, and shape of nanoparticles are understood by Scanning electron microscopy, Energy Dispersive X-Ray Analysis, Fourier-transform infrared spectroscopy, and X-Ray diffraction techniques. A microwave-assisted route is selected for the synthesis of hydroxyapatite nanoparticles to carry out the reaction fast, suppress the enzymatic action, and keep the process environmentally clean and green.

Keywords: Green synthesis; Hydroxyapatite nanoparticles; Psidium guajava leaf extract–Microwave.

1. INTRODUCTION

The process of removing toxic and waste metals in the environment includes microorganisms, plants, and other biological structures, achieved by means of oxidation, reduction, or catalysis of metals with metallic nanoparticles. Metallic nanoparticles produced by biological methods are used in the biomedical field for purposes such as protection from harmful microorganisms, bio-imaging, drug transport, cancer treatment, medical diagnosis and sensor construction because of their unique properties such as being insulator, optics, antimicrobial, antioxidant, anti-metastasis, biocompatibility, stability and manipulability (Frasnelli *et al.* 2017).

Nanomaterials are the mainstay of nanotechnology that serve our lives for many years, can be classified according to their origins, dimensions and structural configurations. Based on their origin, nanomaterials are classified into natural nanomaterials, that are found in nature such as vi-ruses, proteins, enzymes and minerals and artificial nano-materials which are not found in nature and require some processes for their production (Gopi *et al.* 2014). The current research work reveals the synthesis of nanomaterials by green approach. Hydroxyapatite is synthesized with and without guava leaf extract and characterized.

2. MATERIALS & METHODS

2.1 Synthesis of Pure HAp Nanoparticles

The pure HAp nanoparticles were synthesized by taking 3.705g of calcium hydroxide and 2.94g of orthophosphoric acid in separate beakers. Both the precursors are dissolved in the solvent, 50ml of distilled water, under stirring of 30min. After 30min orthophosphoric acid is mixed with calcium hydroxide solution under stirring, a gelatinous white precipitate will be formed. Then NaOH was added to maintain the pH of 12. The solution was aged for 24 hrs at room temperature. Then the precipitate is washed and dried in a microwave oven at 75w for 20 min. The dried sample was grained in mortar. The mixture was kept in a muffle furnace for 4hrs at 400 ^{0}C to get a pure white fine powder.

2.2 Synthesis of capped HAp Nanoparticles

The same procedure was followed in capped Hydroxyapatite synthesis. Here *Psidium Guajava* leaf extract is applied as a solvent instead of distilled water.

3. CHARACTERIZATION TECHNIQUES

3.1 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy (FTIR) is an analytical methodology used in industry and academic laboratories to understand the structure of individual molecules and the composition of molecular mixtures. FTIR spectroscopy uses modulated, mid-infrared energy to interrogate a sample. The infrared light is absorbed in specific frequencies related to the vibrational bond energies of the functional groups present in the molecule. A characteristic pattern of bands is formed, which is the vibrational spectrum of the molecule (Kalaiselvi *et al.* 2017; 2019).

3.2 X-ray Powder Diffraction (XRD)

X-ray diffraction methods are the most effective methods for determining the crystal structure of materials. X-ray diffractometry (XRD), was originally used for examining the crystal structure of powder samples; thus, traditionally, it is called X-ray powder diffractometry (Kalaiselvi *et al.* 2018a; 2018b). Wide-angle X-raydiffraction (WAXD) or wide-angle X-ray scattering (WAXS) is the technique that is commonly used to characterize crystalline structures of polymers and fibers.

3.3 Scanning Electron Microscopy (SEM)

A typical SEM instrument, showing the electron column, sample chamber, EDS detector, electronics console, and visual display monitors. The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample, including external morphology (texture), chemical composition, and crystalline structure, and orientation of materials making up the sample.

The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations (using EBSD). (Kumar *et al.* 2017; Sundrarajan *et al.* 2015) The design and function of the SEM are very similar to the EPMA, and considerable overlap in capabilities exists between the two instruments.

3.4 Energy-Dispersive X-Ray Spectroscopy (EDAX)

Interaction of an electron beam with a sample target produces a variety of emissions, including x-rays. An energy-dispersive (EDAX) detector is used to separate the characteristic x-rays of different elements into an energy spectrum, and EDAX system software is used to analyze the energy spectrum in order to determine the abundance of specific elements (Vasant and Joshi *et al.* 2011; Yuam *et al.* 2017). EDAX can be used to find the chemical composition of materials down to a spot size of a few microns and to create element composition maps over a much broader raster area. Together, these capabilities provide fundamental compositional information for a wide variety of materials.

4. RESULTS & DISCUSSION

4.1 SEM

Scanning electron microscopy is a valuable technique to determine the morphology and particle size of the sample fig.1. Shows SEM image of pure HAp nanoparticles.

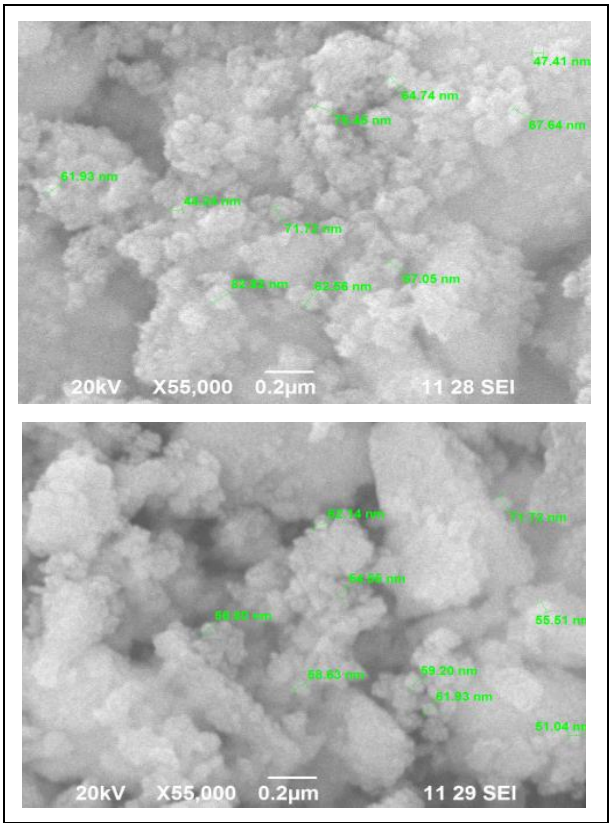

Fig. 1: SEM Images of Pure and Capped Hap

Table 1. XRD Analysis of ZnO Nanoparticles

Element	App Conc	Intensity Corrn	Weight %	Weight % Sigma	Atomic
O K	26.12	0.4387	51.23	0.61	70.08
Na K	2.35	0.6920	2.93	0.19	2.79
P K	19.90	1.3117	13.05	0.26	9.22
Ca K	38.39	1.0070	32.79	0.44	17.91
		Total			100.00

4.2 Energy Dispersive X-Ray Diffraction Analysis

An energy dispersive x-ray (EDX) spectroscopy analysis was performed. The EDX results confirm the obtained material, which is composed of ca and p, as shown in fig. 2(a). The EDAX results confirm the obtained material, which is composed of ca, p and o, as shown in fig. 2(b).

4.3 Fourier Transforms Infrared Spectroscopy

The FTIR spectra predict the characteristic bands present in the sample. The presence of phosphate and

hydroxyl groups is identified, which corresponds to HAp structure. The FTIR spectrum of HAp shows the vibration modes of phosphate at 870.70, 570.82, and 1044.26cm-1.Hydroxyl groups of HAp are revealed at 3454.85 cm-1 and 3744.12 cm-1 . The vibration modes at 569.86, 872.631, and 1043.31cm-1 reveal the presence of phosphate group and Hydroxyl groups at 3456.78 cm-1 and 3636.12 cm-1 for DHAp.

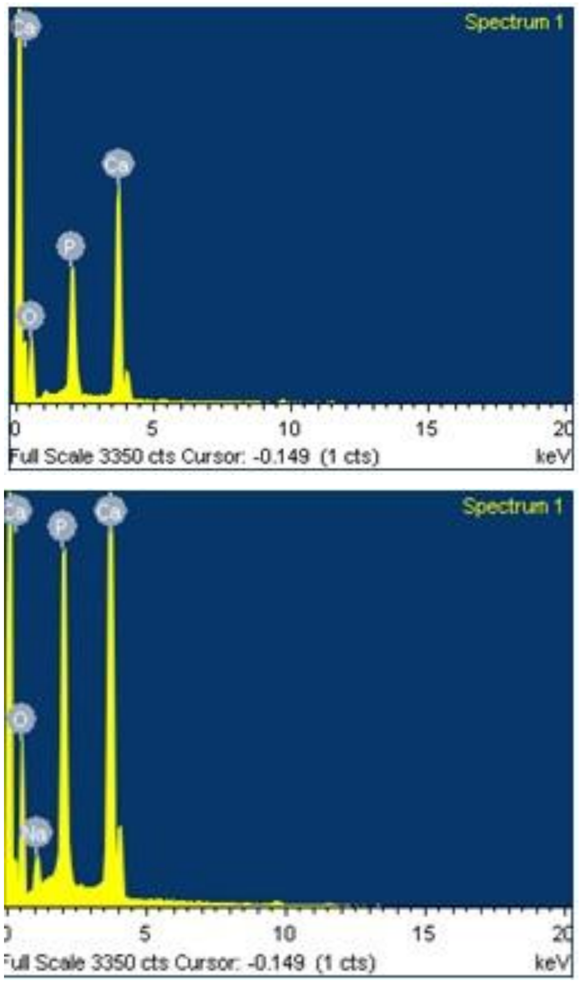

Fig. 2: EDAX Images of Pure and Capped Hap

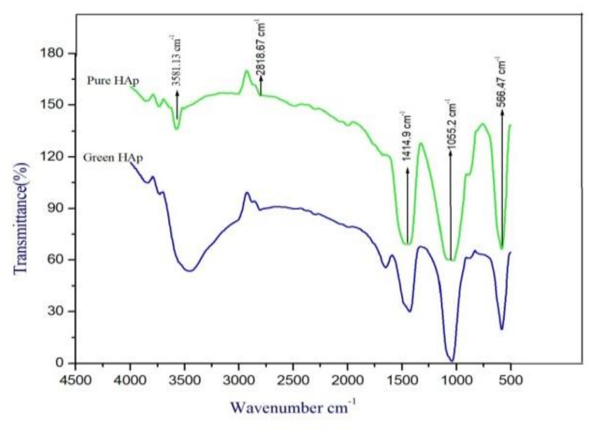

Fig. 3: FTIR Images of Pure and Capped Hap

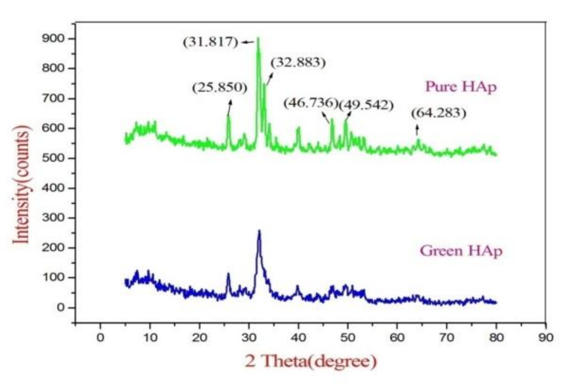

Fig. 4: XRD analyses of pure HAp and D HAp Nanoparticles

4.4 XRD Analysis

The XRD pattern of prepared pure HAp nanoparticles shown in fig. 4. The crystalline size of nanoparticles was determined from the major peak value. The average crystalline calculated by using Debay – Scherer formula is approximately in the range between 5-24nm**.**

Table 2. FTIR Analyses of Pure HAp and D HAp Nanoparticles

S. No.	Sample Name	WAVE NUMBER cm^{-1}				
		O-H stretching vibration	stretching vibration	C=C stretching vibration	CH_3 stretching vibration	P-OStretching vibration
1.	P HAp	3555.67	2918.55	1593.81	1319.58	694.84
2.	D HAp	3538.71	2916.49	1595.07	1317.41	695.18

Table 3. XRD Analyses of Pure HAp and D HAp Nanoparticles

Sample no	2 theta (deg)	FWHM(deg)	D(A^0)	Intensity (counts)	Crystalline size(nm)	Average crystalline size(nm)	hkl	Lattice constant a=b	Lattice constant c	Unit volume
	31.97	0.7205	2.79	226	14.57		201			502.83
P HAp	35.97	0.3467	2.52	11	11.47	15.71	112	9.46	6.89	546.46
	39.10	0.4000	2.30	10	21.11		301			524.98
	32.13	1.32	2.78	128	6.23		112			531.07
D HAp	35.46	0.73	2.52	7	11.37	8.54	301	9.48	6.84	567.14
	39.82	1.05	2.26	21	8.04		212			500.64

5. CONCLUSION

The present study discussed the green synthesis of Hydroxyapatite nanoparticles with and without capping agent *psidium guajava* leaf extract irradiated by Microwave irradiation method. The XRD pattern confirmed the crystalline size of the sample, lattice parameter, and unit cell volumes of nanoparticles are well-matched with the standard value. The crystalline size decreases in DHAp when compared with HAp. The FTIR spectrum reveals the presence of functional groups, and it confirms the phosphate and hydroxyl groups present in the sample. Then the SEM analysis predicts the spherical shaped morphological structure. Finally, EDAX analysis determines the elemental composition and purity of the sample. It confirms by the presence of calcium and phosphate groups.

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